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Megastigmane glycoside from *Ludwigia Stolonifera*

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ABSTRACT

Ludwigia genus belongs to family Onagraceae, is an edible medicinal plant and is also used as a vegetable by the local people in Southwestern China. Some species of this plant, has been used as a traditional treatment for edema, nephritis, and hypertension. Phytochemical study of the CH₂Cl₂: MeOH (1:1) extract of the aerial parts of *Ludwigia stolonifera* afforded a megastigmane glycoside named, roseoside. The structure was determined by comprehensive NMR studies including DEPT, COSY, HMQC, HMBC and MS.

KEYWORDS: *Ludwigia Stolonifera*, megastigmane, glycoside, roseoside.

INTRODUCTION

Ludwigia, (family Onagraceae) from the tribe jussiaea, is an aquatic plant, which is a very variable genus that contains over 80 species grouped in 23 sections (1). Species of this genus is an edible medicinal plant and is also used as a vegetable by the local people in southwestern china (2). Some species of this plant, has been used as a traditional treatment for edema, nephritis, and hypertension (3). Few reports have appeared in the literature on the chemistry and biological activity of this genus; thus previous studies have shown that the crude extract of *Ludwigia octovalvis* possess antidiabetic and immunosuppressive activities (3). Many species of the genus have been investigated and found to contain oleanane-type triterpenes, triterpene acids and flavonoids. This paper describes the isolation, identification and structural elucidation of a megastigmane glycoside from the aerial parts of *Ludwigia stolonifera*.

MATERIALS AND METHODS

General

NMR spectra were measured with a Bruker AMX-500 spectrometer, with TMS as an internal standard. CC: Silica gel (Merck, 60-120 mesh) and Sephadex LH-20 (Pharmacia). TLC and Preparative TLC: Silica gel 60 GF₂₅₄ (Merck). The compound was visualized either by spraying with vanillin reagent or under UV lamp.

Plant material

Ludwigia stolonifera was collected in 2006 from Aswan, South of Egypt, Egypt. A voucher specimen of the collection was identified by Dr. Magdi A. El-Sayed and was deposited in the Department of Botany, Aswan Faculty of Science, Egypt.

Extraction and isolation

Air dried and powdered aerial parts (100 g) of *Cleome arabica* were extracted with CH₂Cl₂-methanol (1:1) at room temperature for 24 h. The extract was concentrated *in vacuo* to give a residue (14 g), which was chromatographed by using flash column chromatography on a silica gel eluted with *n*-hexane-CH₂Cl₂ step-gradient and finally CH₂Cl₂-MeOH (85:15) (3 L each of the solvent). The CH₂Cl₂-methanol fraction (9.5:0.5) was carefully chromatographed on a Sephadex LH-20 column eluted with *n*-hexane-CH₂Cl₂-MeOH (7: 4: 0.5) with increasing the polarity to give a megastigmane glycoside, compound 1 (12 mg).

RESULTS AND DISCUSSION

Compound 1 was obtained from the CH₂Cl₂-MeOH (9.5:0.5 %) fraction and appeared as a dark brown color on the TLC when treated with vanillin-sulphuric acid. Compound 1, was isolated as a white powder, +20.3 (*c* = 0.001, MeOH) and its IR spectrum showed absorption bands at 3250 cm⁻¹ (OH groups) and a conjugated carbonyl group at 1650 cm⁻¹. The EI-MS of 1 gave a molecular ion peak [M]⁺ at *m/z* = 386 and exact mass determination at *m/z* = 386.1941 established the elemental composition as C₁₉H₃₀O₈ (confirmed by ¹³C NMR and DEPT analysis). The fragment ion at *m/z* = 368, due to the elimination of water molecule. The ¹H NMR spectrum showed a broad singlet at δ_H 5.85 (brs, H-4), coupled with a carbon signal at δ_C 127.8 (C-4) in HMQC spectrum. Additionally, it showed a broad singlet signal at δ_H 5.84 integrated for two protons H-7 and H-8, two doublets at δ_H 2.31 (H-2a), 2.14 (H-2b), coupled with a carbon signal at δ_C 51.5, C-2 in HMQC spectrum. Furthermore, the ¹H NMR spectrum revealed the presence of four signals for the methyl groups at δ_H 1.02 (s, H-11), 1.03 (s, H-12), 1.28 (d, *J* = 6.7, H-10), and 1.91 (d, *J* = 1.4, H-13). The presence of the sugar moiety was suggested from the anomeric proton signal at δ_H 4.33 (d, *J* = 7.7, H-1'), which showed a correlation with the double of doublet at δ 3.16 (dd, *J* = 9.1, 7.7, H-2') in ¹H-¹H COSY spectrum. The ¹³C NMR spectrum of 1 indicated the presence of a glucose moiety and thirteen carbon atoms for the aglycone part. With the aid of DEPT and HMQC experiments, the carbons were classified as follows: one carbonyl carbon at δ 201.6 (C-3), four methyl carbons at [δ_C 22.1 (C-10), 25.5 (C-11), 24.3 (C-12), 20.4 (C-13)], two methylene carbons at [δ_C 51.5 (C-2), 63.6 (C-6)], nine methine carbons at [δ_C 127.8 (C-4), 132.1 (C-7), 135.8 (C-8), 78.0 (C-9), 103.4 (C-1'), 76.0 (C-2'), 78.8 (C-3'), 72.4 (C-4'), 78.2 (C-5')], three quaternary carbons at [δ_C 43.2 (C-1), 167.8 (C-5), 80.7 (C-6)].

Confirmation of compound 1 was given by the HMBC analysis, the most important correlations were observed between; H-2_a (δ 2.51, d) and C-1 (δ 43.2), C-11 (δ 25.5), C-12 (δ 24.2); H-2_b (δ 2.14, d) and C-3 (δ 201.6), C-4 (δ 127.8), C-6 (δ 80.7); H-4 (5.85, br s) and C-2 (δ 51.5), C-13 (δ 20.4); H-7 (5.84, br s) and C-6 (δ 80.7), C-8 (δ 135.8), C-9 (δ 78.0); H-8 (5.84, br s) and C-7 (δ 132.1), C-9 (δ 78.0); H-10 (1.28, d) and C-9 (δ 78.0), C-8 (δ 135.8); H-11 (25.5, s) and C-1 (δ 43.2), C-12 (δ 24.2), C-6 (δ 80.7), C-2 (δ 51.5); H-12 (1.03, s) and C-11 (δ 25.5), C-1 (δ 43.2), C-6 (δ 80.7), C-2 (δ 51.5); H-13 (1.91, d) and C-4 (δ 127.8), C-5 (δ 167.8), C-6 (δ 80.7); H-1' (4.33, d) and C-1' (δ 103.4), C-9 (δ 78.0). On the basis of these results, compound 1 was identified as 4-hydroxy-3,5,5-trimethyl-4-[(*E*)-3-(3,4,5-trihydro-pyran-2-yloxy)-but-1-enyl]-cyclohex-2-enone (roseoside), isolated for the first time from *Ludwigia stolonifera* (4).

Table 1: ¹H NMR and ¹³C NMR spectral data of compound 1 (500 MHz, 150 MHz, CD₃OD):-

No.	δ _C	δ _H	HMBC
1	43.2	—	—
2	51.5	a: 2.51 d (17.1) b: 2.14 d (17.1)	H-2b H-2a
3	201.6	—	—
4	127.8	5.85 brs	H3-13
5	167.8	—	—
6	80.7	—	—
7	132.1	5.84 brs	H-9
8	135.8	5.84 brs	H-9
9	78.0	4.41 m	H-10, H-7, H-8
10	22.1	1.25 d (6.7)	H-9
11	25.5	1.02 s	—
12	24.2	1.03 s	—
13	20.4	1.91 d (1.4)	H-4
1'	103.4	4.33 d (7.7)	—
2'	76.0	3.16 dd (9.1, 7.7)	—
3'	78.8	3.33 t (9.1)	H-1'
4'	72.4	3.25 dd (9.7, 9.1)	H-1'
5'	78.2	3.22 m	H-3'
6'	63.6	a: 3.84 dd (11.9, 2.0)	H-4'

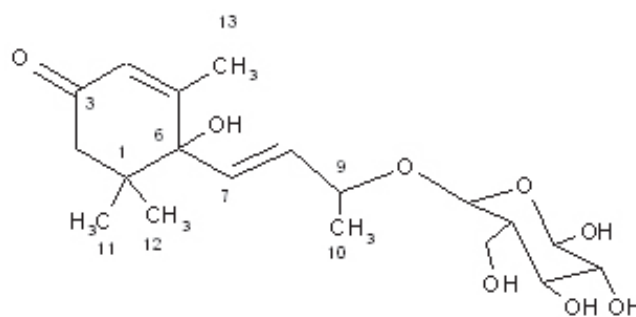


Figure 1: Structure of roseoside.

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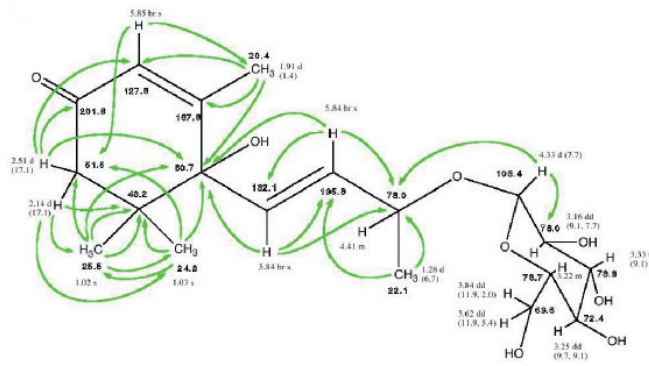


Figure 2: HMBC of roseoside

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